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MORPHOLOGY OF OXIDIC LAYERS PREPARED BY SOL-GEL

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ABSTRACT: Thin layers of titanium dioxide, zirconium dioxide and silicon dioxide are extensively studied because of their interesting chemical, electrical and optical properties. These layers were obtained in sol-gel process and prepared as thin layers on microscope slide glasses by dip-coating method. In order to define the influence of modification on surface properties (e.g., roughness) and morphology, various types of precursors were used. The topography was investigated with the use of atomic force microscopy (AFM).

KEY WORDS: oxidic layers, sol-gel, AFM, topography

1. INTRODUCTION

Many glass properties are more or less strongly controlled by the surface state, i.e., the micro- or nanoscale morphologies. Of the techniques developed both to modify and to improve glass surfaces, the sol-gel method is one of the most attractive and effective ones [1].

Sol-gel layers prepared on the surface of glass affect favorably the properties of the glass substrate, thus expanding the possibilities of utilizing glass in optics, communication technology, electronics and other additional fields of technology. The inorganic sol-gel layers may also be used to protect the glass surface from the effects of aggressive solutions. Their performance in this respect depends primarily on their chemical composition, porosity and thickness of the layer. Layers containing TiO_2, ZrO_2 and SiO_2 exhibit a very good resistance to atmospheric effects and to chemical effects [2].

The atomic force microscope (AFM) has proven to be an essentially valuable tool for investigating glass surfaces and surfaces of coatings [3]. Recently the crystallization and the corrosion mechanisms of TiO_2 coatings on glass substrates were studied, and it was demonstrated that atomic force microscopy (AFM) is ideally suited for such an investigation [1]. The atomic force microscopy (AFM) is a complex instrument used to investigate the surface at the micro- and nanometer scale [4, 5].

2. EXPERIMENTAL

TiO_2, ZrO_2 and SiO_2 layers were prepared on microscope slide glasses by dip-coating method from correspondent sols [6]. Composition of sols is present in Tab.1. The thicknesses of layers were calculated from reflectance values on the assumption of normal incidence of monochromatic light on layer [7, 8] and are present in Tab.1.

The surfaces of layers were observed by the non-contact regime AFM NT-206. Valuation of surfaces was made on the base of statistic characteristics of the profile-The Medium Arithmetic Variation of Ra Profile, Angularity Rsk, Sharpness Rku.

Tab.1 Composition of sols and thicknesses of layers

layer thickness	component	*n / mol*
TiO_2 ~ 50 nm	$Ti(PrO)_4$	*1*
	H_2O	*2.2*
	izopropylalcohol	*30*
	HNO_3	*0.76*
ZrO_2 ~ 40 nm	$Zr(PrO)_4$	*1*
	acetylacetone	*7,8*
	H_2O	*26*
	izopropylalcohol	*26.5*
	HNO_3	*0,4*
SiO_2 ~ 90 nm	$Si(EtO)_4$	*1*
	acetic acid anhydride	*1*
	H_2O	*14.3*
	izopropylalcohol	*0.105*
	HNO_3	*4*

3. RESULTS AND DISCUSSION

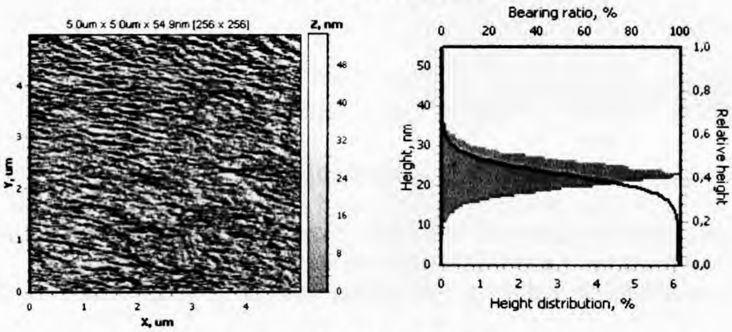

Fig. 1: AFM image of clean microscope slide glasses

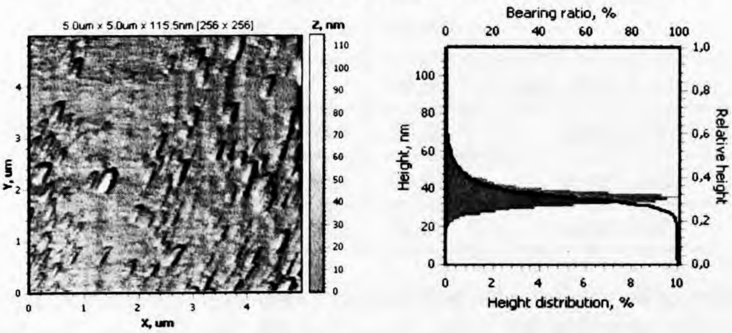

Fig. 2: AFM image of TiO_2 layer

On the Fig. 1 we see, that the surface of clean glass is covered by elongated spur. These formations have different parameters and various concentrations on surface samples.

On the Fig. 2 we see clusters, which are together connecting. The shape of clusters is non-uniform. The surface of these clusters is uneven.

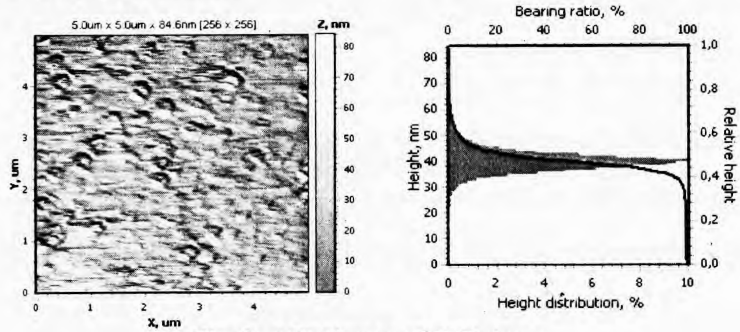

Fig. 3: AFM image of ZrO_2 layer

On the Fig. 3 we see clusters, which are separate. The shape of clusters is non-uniform. The surface of these clusters is uneven.

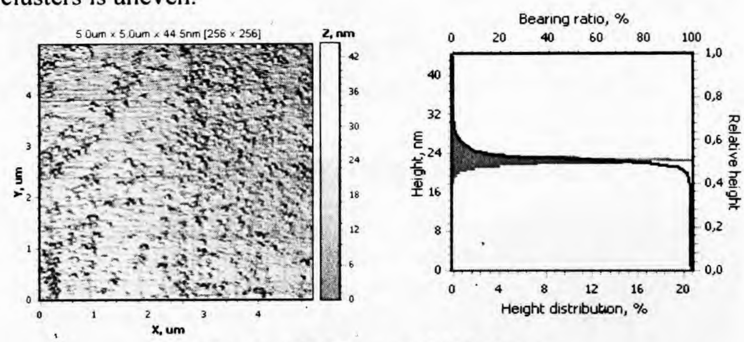

Fig. 4: AFM image of SiO_2 layer

On the Fig. 1 we see, that the surface of clean glass is covered by elongated spur. These formations have different parameters and various concentrations on surface samples.

In Tab. 2 are presented statistical characteristic of samples profile obtained from histograms.

Tab.2: Statistical characteristics of sample's surface

	Hmean	Ra	Rq	Rsk	Rku
microscope slide glasses	36,64	5,25	7,75	1,94	11,0
TiO_2 layer	22,72	3,19	4,23	0,12	4,86
ZrO_2 layer	40,10	3,56	5,33	1,02	9,98
SiO_2 layer	22,82	1,04	1,74	1,32	16,07

From the comparison of Ra values for the pure glass and each layer it is obvious, that the surface is being exterminated while spreading the layer. It is observed, that Ra value decreases with the layer's thickness.

It results from the Ra values comparison that all profiles have partition with elongated right end, this means that they have got larger variations rather than smaller ones, more higher peaks. TiO_2 layer is different from the others, it approaches normal division more.

It results from Rsk values, that all profiles have leptocurtic divisions, this means that they have higher peaks, what we can interpret as an increased multitude of the one of the variations. It is obvious from the Rku values and the histograms that SiO_2 layer has got the narrowest division of heights, ZrO_2 layer has got wider division and this can be compared with pure glass, the widest division has got the TiO_2 layer.

4. CONCLUSION

We have observed the TiO_2, ZrO_2 and SiO_2 layers' surfaces prepared by the sol-gel method using the AFM. On the base of the statistic elaboration of gathered results we can point out, that the layers spread on the glass substrate cause extermination. Rate of this extermination is indirectly proportionate to the layer's thickness. According to the Rku values and histograms we can point out that the pure glass, SiO_2 and ZrO_2 layers have got approximately equal objects on their surfaces (the leptocurtic division), while the TiO_2 layer has got the heights division close to the normal division.

5. REFERENCES

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